इंटरनेट मानक

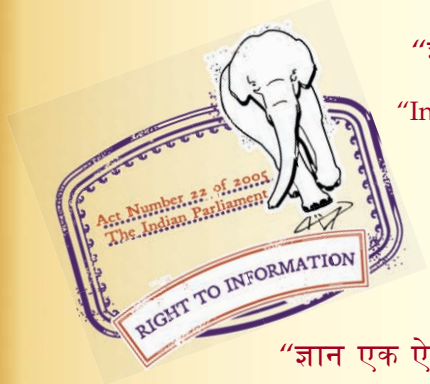
Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 14794 (2000): Isobutene-Isoprene Rubber (IIR) - Evaluation Procedures [PCD 13: Rubber and Rubber Products]



"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

IS 14794 : 2000
ISO 2302 : 1995

भारतीय मानक

आइसोब्यूटीन–आइसोप्रीन रबड़ (आई आई आर) — मूल्यांकन प्रक्रियाएँ

Indian Standard

ISOBUTENE-ISOPRENE RUBBER (IIR) — EVALUATION PROCEDURES

ICS 83.060

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 2000

Price Group 4

NATIONAL FOREWORD

This Indian Standard which is identical with ISO 2302 : 1995 'Isobutene-isoprene rubber (IIR) — Evaluation procedures' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Rubber Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO standard has been proposed to be approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 37 : 1994 Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties	IS 3400 (Part 1) : 1987 Methods of test for vulcanized rubbers : Part 1 Tensile stressstrain properties (*second revision*)	Technically equivalent with minor deviations
ISO 247 : 1990 Rubber — Determination of ash	IS 3660 (Part 3) : 1988 Methods of test for natural rubber : Part 3 Determination of ash (NR : 3) (*second revision*)	do
ISO 248 : 1991 Rubbers, raw — Determination of volatile — Matter content	IS 3660 (Part 2) : 1985 Methods of test for natural rubber : Part 2 Determination of volatile matter (*second revision*)	do
ISO 289-1 : 1994 Rubber, unvulcanized — Determinations using a shearing-disc viscometer—Part 1 : Determination of Mooney viscosity	IS 3660 (Part 7) : 1988 Methods of test for natural rubber : Part 7 Determination of Mooney viscosity (NR : 8) (*second revision*)	do
ISO 471 : 1995 Rubber — Temperatures, humidities and times for conditioning and testing	IS 13867 : 1993 Rubber standard temperatures, humidities and times for the conditioning and time interval between vulcanization and testing of test pieces	do

(*Continued on third cover*)

Indian Standard

ISOBUTENE-ISOPRENE RUBBER (IIR) — EVALUATION PROCEDURES

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, a standard test formulation, and equipment and processing methods for evaluating the vulcanization characteristics of all types of isobutene-isoprene rubber (IIR).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*

ISO 247:1990, *Rubber — Determination of ash.*

ISO 248:1991, *Rubbers, raw — Determination of volatile-matter content.*

ISO 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity.*

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

ISO 1795:1992, *Rubber, raw, natural and synthetic — Sampling and further preparative procedures.*

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 6502:1991, *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

3 Sampling and further preparative procedures

A laboratory sample of approximately 1,5 kg shall be taken by the method described in ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Prepare a test portion, without milling, in accordance with the preferred procedure in ISO 1795.

Determine the Mooney viscosity in accordance with ISO 289-1 on a test portion cut directly from the laboratory sample and as free as possible from air and pockets that may trap air against the rotor and die surface.

Because the Mooney viscosity of high-molecular-mass isobutene-isoprene rubbers does not vary linearly with their molecular mass, it is necessary to use different test temperatures for high- and low-Mooney rubbers. For low-Mooney rubbers (i.e. those not exceeding 60 under the conditions given here), the viscosity shall be determined as ML (1 + 8) at 100 °C.

For high-Mooney rubbers, the viscosity shall be determined as ML (1 + 8) at 125 °C.

4.2 Volatile matter

Determine the volatile-matter content by the hot-mill method or by the oven method as specified in ISO 248.

4.3 Ash

Determine the ash in accordance with method A or method B in ISO 247:1990.

5 Preparation of test mixes for evaluation of isobutene-isoprene rubbers

5.1 Standard test formulation

The standard test formulation is given in table 1.

The materials shall be national or international standard reference materials (or as agreed by the interested parties).

Table 1 — Standard test formulation for evaluation of isobutene-isoprene rubbers

Material	Parts by mass
Isobutene-isoprene rubber (IIR)	100,0
Stearic acid	1,00
Current industry reference black	50,00
Zinc oxide	3,00
Sulfur	1,75
TMTD[1)]	1,00
Total	156,75

1) Tetramethylthiuram disulfide.

5.2 Procedure

5.2.1 Equipment and procedure

The equipment and procedure for the preparation, mixing and vulcanization shall be in accordance with ISO 2393.

Three alternative mixing procedures are specified:

Method A — Mill mixing

Method B — Internal mixer mixing

Method C — Miniature internal mixer mixing.

Method B is presented as informative annex A since insufficient experience has been gained with this method to include it as part of the standard.

NOTE 1 These procedures may not give identical results.

5.2.2 Method A — Mill mixing procedure

The standard laboratory-mill batch mass, in grams, shall be based on four times the formulation mass (i.e. 4 × 156,75 g = 627 g). The surface temperature of the rolls shall be maintained at 45 °C ± 5 °C throughout the mixing.

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

A mill batch mass based on two times the formulation mass may also be used, but, in this case, more adjustments to the mill openings will be necessary.

	Duration (min)
a) Band the rubber with the mill opening set at 0,65 mm.	1,0
b) Mix the carbon black and the stearic acid and add evenly across the rolls at a uniform rate. Increase the mill opening at intervals to maintain a constant rolling bank. When all the black has been incorporated, make one 3/4 cut from each side. Be certain to add all the black that has dropped into the mill pan.	10,0
c) Add the zinc oxide, the sulfur and the TMTD.	3,0
d) Make three 3/4 cuts from each side.	3,0
e) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls six times.	2,0
Total time	19,0

f) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for curemeter testing.

g) Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37.

h) Condition the batch for 2 h to 24 h, after mixing and prior to vulcanizing, if possible at standard laboratory temperature and humidity as defined in ISO 471.

5.2.3 Method C — Miniature internal mixer procedure

For a miniature internal mixer having a nominal mixing capacity of 64 cm^3, a batch mass corresponding to 0,46 times the formulation mass (i.e. 0,46 × 156,75 g = 72,10 g) has been found to be suitable.

Mix with the head temperature of the miniature internal mixer maintained at 60 °C ± 3 °C and the unloaded-rotor speed at 6,3 rad/s to 6,6 rad/s (60 rpm to 63 rpm).

Prepare the rubber by passing it once through a mill with the temperature set at 50 °C ± 5 °C and an opening of 0,5 mm. Cut the sheet into strips that are 25 mm wide.

NOTE 2 Compounding materials other than rubber, e.g. carbon black, may be added to miniature internal mixer batches more precisely and with greater ease if they are previously blended together in proportion to the mass required by the formulation. Such blends may be made using one of the following.

a) A mortar and pestle.

b) A biconical mixer. Agitate for 10 min with the intensifier bar turning.

c) A blender. Mix for five periods of 3 s each, scraping the inside of the blender to dislodge materials stuck to the sides after each 3 s period. (A "Waring"-type blender has been found suitable for this method.) CAUTION — If mixed longer than 3 s at a time, the stearic acid may melt, thus preventing good dispersion.

	Duration (min)	Cumulative time (min)
a) Load the rubber, lower the ram and allow the rubber to be masticated.	1,0	1,0
b) Raise the ram and add the zinc oxide, sulfur, stearic acid and TMTD, taking care to avoid any loss. Then add the carbon black, sweep the orifice and lower the ram.	1,0	2,0
c) Allow the batch to mix.	3,0	5,0

d) Turn off the rotor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature.

The final temperature of the batch discharged after 5 min shall not exceed 120 °C. If necessary, adjust the batch mass or the head temperature so that this condition is achieved.

e) Pass the batch through a mill set at 50 °C ± 5 °C twice at a 3,0 mm mill opening.

f) Check the batch mass (see ISO 2393) and record. If it differs from the theoretical value by more than 0,5 %, discard the batch and remix.

g) Cut a test piece for testing vulcanization characteristics in accordance with ISO 3417 or ISO 6502, if required. Condition the test piece for 2 h to 24 h at 23 °C ± 3 °C before testing.

h) If required, sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ring test pieces in accordance with ISO 37. To obtain the effects of mill direction, pass the folded batch four times between mill rolls set at 50 °C ± 5 °C and at the appropriate mill opening. Cool on a flat, dry surface.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard laboratory temperature and humidity as defined in ISO 471.

6 Evaluation of vulcanization characteristics

6.1 Using an oscillating-disc curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1}, $t'_c(50)$ and $t'_c(90)$

in accordance with ISO 3417, using the following test conditions:

oscillation frequency:	1,7 Hz (100 cycles per minute)
amplitude of oscillation:	1° arc An amplitude of oscillation of 3° arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1}.
selectivity:	To be chosen to give at least 75 % of full-scale deflection at M_H.
die temperature:	160 °C ± 0,3 °C
pre-heat time:	None

6.2 Using a rotorless (torsion shear) curemeter

Measure the following standard test parameters:

F_L, F_{max} at defined time, t_{s1}, $t'_{0,50}$ and $t'_{0,90}$

in accordance with ISO 6502, using the following test conditions:

oscillation frequency:	1,7 Hz (100 cycles per minute)
amplitude of oscillation:	0,5° arc An amplitude of oscillation of 1° arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1}.
selectivity:	To be chosen to give at least 75 % of full-scale deflection at F_{max}.
die temperature:	160 °C ± 0,3 °C
pre-heat time:	None

NOTE 3 The two types of curemeter may not give identical results.

7 Evaluation of tensile stress-strain properties of vulcanized test mixes

Vulcanize sheets at 150 °C for 20 min, 40 min and 80 min.

Condition the vulcanized sheets for 16 h to 96 h, at a standard laboratory temperature, and, if possible, at a standard laboratory humidity defined in ISO 471.

Measure the stress-strain properties in accordance with ISO 37.

NOTE 4 Method C provides sufficient compounded material for evaluation of vulcanization characteristics by a curemeter test and the evaluation of stress-strain properties on one vulcanized sheet. The recommended vulcanization time is 40 min at 150 °C, but other values may be appropriate.

8 Test report

The test report shall include the following:

a) a reference to this International Standard;

b) all details necessary for the identification of the sample;

c) the temperature used for the Mooney viscosity determination;

d) the method used for the volatile-matter-content determination (mill or oven);

e) the method used for the ash determination (method A or B of ISO 247:1990);

f) the reference materials used;

g) the mixing procedure used;

h) the mill batch factor used in 5.2.2;

i) the conditioning environment used in 5.2.2 h), 5.2.3 i), clause 7 and A.1 i);

j) in clause 6:

— the type of curemeter used and the reference standard,

— the time for M_H or F_{max} and

— the amplitude of oscillation used for the curemeter test;

k) any unusual features noted during the determination;

l) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;

m) the results and the units in which they have been expressed;

n) the date of the test.

Annex A
(informative)

Method B — Internal mixer for initial and mill for final mixing

For an internal mixer of Type A1 (see ISO 2393) having a nominal capacity of 1 170 cm^3 ± 40 cm^3, a batch mass corresponding to 8,5 times the formulation mass (i.e. 8,5 × 156,75 g = 1332 g) has been found to be suitable.

The speed of the fast rotor shall be set at 7 rad/s to 9 rad/s (67 rpm to 87 rpm).

The final temperature of the batch discharged after 5 min mixing time shall be between 150 °C and 170 °C. If necessary, adjust the batch mass to achieve the mixing conditions specified.

During final mixing, a good rolling bank at the nip of the rolls shall be maintained. If this is not attained with the nip settings specified, small adjustments to the mill openings may be necessary.

A.1 Stage 1 — Initial mixing procedure

	Duration (min)	**Cumulative time** (min)
a) Adjust the temperature of the internal mixer to a starting temperature of 50 °C. Close the discharge door, start the rotors and raise the ram.		
b) Load the rubber, lower the ram and allow the rubber to be masticated.	0,5	0,5
c) Raise the ram, load the zinc oxide, stearic acid and carbon black, and lower the ram.	0,5	1,0
d) Allow the batch to mix.	2,0	3,0
e) Raise the ram, clean the mixer throat and the top of the ram, and lower the ram.	0,5	3,5
f) Allow the batch to mix.	1,5	5,0
g) Discharge the batch.		
Total time	5,0	

h) Immediately check the temperature of the batch with a suitable measuring device. If the temperature as measured falls outside the range 150 °C to 170 °C, discard the batch. Pass the batch three times through a mill with a mill opening of 2,5 mm and a temperature of 50 °C ± 5 °C. Sheet the batch to an approximate thickness of 10 mm and check-weigh the batch (see ISO 2393). If the mass differs from the theoretical value by more than 0,5 %, discard the batch and re-mix.

i) Leave the batch for at least 30 min and up to 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 471.

A.2 Stage 2 — Final mill mixing procedure

a) The standard laboratory-mill batch mass, in grams, shall be based on three times the formula mass (462 g masterbatch).

b) Set the mill temperature at 50 °C ± 5 °C and the mill opening to 1,5 mm.

	Duration (min)	**Cumulative time** (min)
c) Band the masterbatch on the slow roll.	1,0	1,0
d) Add the sulfur and the TMTD. Do not cut the band until the sulfur and accelerator are completely dispersed.	1,5	2,5
e) Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,5	5,0

f) Cut the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternatively. 2,0 7,0

Total time 7,0

g) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for curemeter testing.

h) Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37. Check the batch mass and record. If it differs from the theoretical value by more than 0,5 %, discard the batch and re-mix.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard laboratory temperature and humidity as defined in ISO 471.

(*Continued from second cover*)

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 1795 : 1992 Rubber, raw natural and synthetic — Sampling and further preparative procedures	IS 5599 : 1999 Rubber raw — Identical natural and synthetic — Methods of sampling and sample preparation (*first revision*)	Technically equivalent with minor deviations
ISO 2393 : 1994 Rubber test mixes — Preparation mixing and vulcanization — Equipment and procedures	IS 3660 (Part 8) Methods of test for natural rubber : Part 8 Mixing and vulcanizing in standard compound (NR : 9) (*second revision*)	do
ISO 3417 : 1991 Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter	Nil	
ISO 6502 : 1991 Rubber — Measurement of vulcanization characteristics with rotorless curemeters	Nil	

In the case of ISO 3417 : 1991 and ISO 6502 : 1991, the Technical Committee responsible for the preparation of this standard has reviewed their contents and has decided that they are acceptable for use in conjunction with this standard.

For tropical countries like India, the standard temperature and the relative humidity shall be taken as 27 ± 2°C and 65 ± 5 percent respectively.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 14 (1432).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 323 01 31, 323 94 02, 323 33 75

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices:	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17, 323 38 41
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	337 84 99, 337 85 61, 337 86 26, 337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43, 60 20 25
Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600113	235 02 16, 235 04 42, 235 15 19, 235 23 15
Western : Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093	832 92 95, 832 78 58, 832 78 91, 832 78 92

Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PATNA. PUNE. THIRUVANANTHAPURAM.

Printed at New India Printing Press, Khurja, India